

FORM-PTO-1390
(Rev. 12-29-99)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

022701-915

U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.51)

09/720598
Unassigned

INTERNATIONAL APPLICATION NO.

PCT/FR99/01524

INTERNATIONAL FILING DATE

24 June 1999

PRIORITY DATE CLAIMED

25 June 1998

TITLE OF INVENTION

METHOD FOR EVAPORATING AMINONITRILE

APPLICANT(S) FOR DO/EO/US

Gérald BOCQUENET, Henri CHIARELLI and Philippe LECONTE

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than national examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and the PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
 - ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:
 - 1) International Preliminary Examination Report
 - 2) International Search Report



U.S. APPLICATION NO. (If known, see 37 CFR 1.50)

Unassigned

09/720598

INTERNATIONAL APPLICATION NO
PCT/FR/99/01524ATTORNEY'S DOCKET NUMBER
022701-91517. ☒ The following fees are submitted:

CALCULATIONS

PTO USE ONLY

Basic National Fee (37 CFR 1.492(a)(1)-(5)):

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO \$1,000.00 (960)

International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO \$860.00 (970)

International preliminary examination fee (37 CFR 1.482) not paid to USPTO
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$710.00 (958)

International preliminary examination fee paid to USPTO (37 CFR 1.482)
but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$690.00 (956)

International preliminary examination fee paid to USPTO (37 CFR 1.482)
and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00 (962)

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$ 860.00

Surcharge of **\$130.00 (154)** for furnishing the oath or declaration later than
months from the earliest claimed priority date (37 CFR 1.492(e)). 20 ☐ 30 ☐

\$ 0.00

Claims	Number Filed	Number Extra	Rate	
Total Claims	13 -20 =	0	X \$18.00 (966)	\$ 0.00
Independent Claims	1 -3 =	0	X \$80.00 (964)	\$ 0.00
Multiple dependent claim(s) (if applicable)			+ \$270.00 (968)	\$ 0.00

TOTAL OF ABOVE CALCULATIONS =

\$ 860.00

Reduction for 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be
filed. (Note 37 CFR 1.9, 1.27, 1.28).

\$ 0.00

SUBTOTAL =

\$ 860.00

Processing fee of **\$130.00 (156)** for furnishing the English translation later than
months from the earliest claimed priority date (37 CFR 1.492(f)). 20 ☐ 30 ☐

\$ 0.00

TOTAL NATIONAL FEE =

\$ 860.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by
an appropriate cover sheet (37 CFR 3.28, 3.31). **\$40.00 (581)** per property +

\$ 0.00

TOTAL FEES ENCLOSED =

\$ 860.00

Amount to be:
refunded \$

charged \$

- a. ☐ A check in the amount of \$_____ to cover the above fees is enclosed.
- b. ☒ Please charge my Deposit Account No. 02-4800 in the amount of \$ 860.00 to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Norman H. Stepano, Esquire
BURNS, DOANE, SWECKER & MATHIS, L.L.P.
P.O. Box 1404
Alexandria, Virginia 22313-1404
(703) 836-6620

SIGNATURE

Matthew L. Schneider

NAME

Date: December 26, 2000

32,814

REGISTRATION NUMBER

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of)	
)	
Gérald BOCQUENET et al.)	Group Art Unit: Unassigned
)	
Application No.: Corresponds to International)	Examiner: Unassigned
Application No. PCT/FR99/01524)	
)	
International Application Filed: June 24, 1999)	
)	
For: METHOD FOR EVAPORATING)	
AMINONITRILE)	

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D. C. 20231

Sir:

Preliminary to examination of the above-identified application, kindly amend the application in the following manner.

IN THE CLAIMS:

Kindly amend Claims 3-9 and 13 in the following manner.

Claim 3, line 1: delete "claims 1" and insert therefor -- claim 1, --; and

line 2: delete "and 2,".

Claim 4, line 1: delete "claims 1 to" and insert therefor -- claim 1, --; and

line 2: delete "3,".

Claim 5, line 1: delete "claims 1 to" and insert therefor -- claim 1 --; and
line 2: delete "4,".

Claim 6, line 1: delete "claims 1 to" and insert therefor -- claim 1 --; and
line 2: delete "5,".

Claim 7, line 1: delete "claims 1 to" and insert therefor -- claim 1 --; and
line 2: delete "6,".

Claim 8, line 1: delete "claims 1 to" and insert therefor -- claim 1 --; and
line 2: delete "7,".

Claim 9, line 1: delete "claims 1 to" and insert therefor -- claim 1 --; and
line 2: delete "8,".

Claim 13, line 1: delete "claims 1 to" and insert therefor -- claim 1 --; and
line 2: delete "12,".

REMARKS

By way of the foregoing amendments to the claims, Claims 3-9 and 13 have been amended to delete the multiple dependency.

Early and favorable consideration with respect to this application is respectfully requested.

Respectfully submitted,

BURNS, DOANE, SWECKER, & MATHIS, L.L.P.

By: Matthew L. Schneider

Matthew L. Schneider
Registration No. 32,814

P. O. Box 1404
Alexandria, Virginia 22313-1404
Phone: (703) 836-6620

Date: December 26, 2000

PROCESS FOR VAPORIZING AMINONITRILE

The present invention relates to the vaporization of aminonitrile and water under conditions which limit or eliminate the formation of heavy by-products, especially aminocarboxylic acid oligomers.

The reaction between an aminonitrile and water results in the formation of lactam, especially caprolactam when 6-aminocapronitrile is employed.

This reaction can be carried out in liquid phase with heating and under elevated pressure. It can also be carried out in vapour phase. For this second embodiment, it is therefore necessary to convert the aminonitrile and the water to the vapour state. For examples of carrying out aminonitrile hydrolysis in vapour phase reference may be made, in particular, to the patent EP-A-0 659 741 and the international application WO-A-96/22974.

The choice of the manner of vaporization of the aminonitrile and water is not trivial.

In fact, it is possible to consider forming a liquid water/aminonitrile mixture and then heating this mixture to a temperature sufficient to vaporize the two components. In this case, the formation of heavy compounds having an amide function or carboxylic salt function (oligomers) is observed. These compounds are capable of attaching themselves, at least in part, to the catalyst and so reducing its service life.

Furthermore, they deposit in the apparatus, and foul it. This necessitates periodic cleaning of the said apparatus and hence the relatively frequent shutdown of the plant, with all the economic consequences, as can be imagined.

Another technique which could be considered would be to vaporize the streams of aminonitrile and water separately. The Applicant has noted that, at the temperatures required to vaporize the aminonitrile, it undergoes decomposition, in considerable proportions, to give a compound of the amidine or polyamidine type (condensation of two or more molecules of aminonitrile with elimination of ammonia).

In order to avoid these various drawbacks, a process for vaporizing aminonitrile and water has now been found which is characterized in that the water, in the vapour state, is used as the carrier gas for this vaporization.

The decomposition of the aminonitrile depends on the evaporation temperature and on the residence time of the liquid during its evaporation. In the process of the invention, therefore, the residence time of the liquid is minimized by the technology of the evaporator and the water in the vapour state reduces the partial pressure of the aminonitrile, so lowering its evaporation temperature.

The molar ratio of water to aminonitrile can vary greatly in the process of the invention. It

depends essentially on the cyclizing hydrolysis process in which the reactants will be deployed. This molar ratio between the water and aminonitrile employed is commonly between 0.5 and 100 and preferably between 1 and 20. The upper value of this ratio is not critical for the invention, although higher ratios are of virtually no interest for the hydrolysis reaction on economic grounds.

Generally, the water vapour will be at a temperature of from 120 to 600°C and preferably from 200 to 550°C.

The aminonitrile will generally be employed at a temperature of from 20 to 300°C. Preferably, this temperature will be from 100 to 250°C.

The aminonitrile/water vapour mixture is brought rapidly in a heat exchanger to a temperature at which the vaporization of the mixture is complete.

If appropriate, this temperature can be that at which the reaction between the aminonitrile and water will be conducted. Such a reaction temperature is commonly between 200 and 450°C and preferably between 250 and 400°C.

The absolute pressure at which the vaporization of the aminonitrile is conducted is generally from 0.1 to 3 bar.

The aminonitrile employed in the process of the invention is more particularly a linear or branched aliphatic aminonitrile having 3 to 12 carbon atoms.

By way of examples, mention may be made more particularly of the aliphatic aminonitriles originating from the hydrogenation to a primary amine function of one of the two nitrile functions of dinitriles such as

5 adiponitrile, methylglutaronitrile,
ethylsuccinonitrile, dimethylsuccinonitrile,
malononitrile, succinonitrile, glutaronitrile and
dodecanedinitrile.

The most important aminonitrile is

10 6-aminocapronitrile, the cyclizing hydrolysis of which
leads to caprolactam, whose polymerization yields nylon
6.

For convenience, in the text below, reference may be made more particularly to 6-aminocapronitrile

15 (or ACN).

The process is performed with the aid of a system without retention of liquid.

The technologies which can be employed in order to limit the residence time of the product in

20 liquid phase during the evaporation are of two types:

- evaporation of the aminonitrile as a film on a heated surface;
- evaporation of an at least partly liquid mist of aminonitrile in the superheated water vapour; in this

25 case, the instances of contact between liquid and hot wall are replaced by contact between gas and liquid droplets.

In the case of evaporation as a film on a

heated surface, the heat required for the evaporation is supplied on the one hand by the appreciable heat of the vapour and of the aminonitrile and on the other hand by transfer of heat across the evaporating surface. The evaporator is of the falling-film evaporator type.

The liquid can be distributed over the tubes of the evaporator in accordance with the distribution systems generally employed in this type of technology:

- supply of the at least partly liquid aminonitrile to the tube plate, then distribution of this aminonitrile in each tube;
- distribution of the at least partly liquid aminonitrile in each tube by atomization to a mist of the aminonitrile above the tube plate; this technology has the advantage over the preceding one of reducing further the residence time in liquid phase at high temperature; the liquid can be atomized by means of a nozzle which is fed with the liquid alone or, better still, by a nozzle which is fed simultaneously with the liquid and the water vapour.

In the case of evaporation in a mist with contact between gas and water droplets, all of the heat is supplied by the appreciable heat of the two components, the aminonitrile, which is at least partly in liquid phase, and the water in vapour phase.

The temperature of the water vapour and the temperature of the at least partly liquid aminonitrile

are selected such that the mist obtained is at a temperature which is either equal to or greater than the dew point of the water/aminonitrile mixture which makes up the said mist. Obviously, the dew point
5 depends on the ratio of water to aminonitrile and is easily determined for the selected ratio.

Thus, by way of example, at atmospheric pressure, the dew point is 180°C for a water/6-amino-capronitrile (ACN) ratio of 4, 110°C for a water/ACN
10 molar ratio of 56, 210°C for a water/ACN ratio of 1, and 230°C for pure ACN.

This evaporation in a mist by contact between gas and liquid droplets can be single-stage or multistage. If the evaporation is single-stage, the
15 temperatures of the aminonitrile and of the water vapour are such that the vaporization of the liquid can be total or partial. If the evaporation is multistage, the stream of aminonitrile, preheated to 230°C, for example, is divided into a number of portions, three or
20 four; the first portion of this liquid is mixed with the superheated water vapour, at 300°C for example, such that all of the liquid is vaporized, the temperature of the mixture reducing simultaneously to around the dew point as a consequence of the
25 vaporization. The mixture in the vapour state is subsequently superheated, to 300°C for example, and then mixed again with the second portion of the liquid, which vaporizes in turn; the procedure is repeated for

as many times as is necessary to obtain the total vaporization of the liquid. In this process, the mist of liquid is generated at each stage by means of atomizing nozzles, the mixture being subsequently made
5 in a volume sufficient to ensure the total evaporation of the liquid.

The system for vaporizing the aminonitrile will preferably be selected such that the dwell time of liquid aminonitrile in the said system, comprising the
10 preheating of the said aminonitrile, is less than or equal to one minute, preferably less than or equal to 5 seconds.

The examples which follow illustrate the present invention.

15 EXAMPLE 1

200 g/h of 6-aminocapronitrile (ACN), preheated to 230°C, and 129 g/h of water vapour at 300°C are injected through a 1 mm nozzle.

The mist thus formed is vaporized, then
20 superheated to 300°C with the aid of an exchanger before it supplies a hydrolysis reactor containing 162 g of alumina, the said reactor being maintained at 300°C.

Over more than 400 h of operation, no reactor
25 fouling nor any reduction in catalytic activity (measured by the rate of conversion of ACN at a constant flow rate equal to 99%) was observed.

COMPARATIVE TEST 1

329 g/h of an ACN/water mixture containing 61% by weight ACN are fed into a 200 ml evaporator heated at 300°C.

- 5 The gaseous mixture emerging from the evaporator is passed to a hydrolysis reactor containing 162 g of alumina, the said reactor being maintained at 300°C.

The test is stopped after 172 h of operation.

- 10 During this period, the rate of conversion of ACN has passed from 99% to 95%.

- Following disassembly of the apparatus, the presence of a solid (nylon 6) is noted inside the evaporator and at the entrance of the hydrolysis
15 reactor (25% of the height of the contents of the said reactor have solidified).

CLAIMS

1. Process for vaporizing aminonitrile and water, characterized in that the water in the vapour state is used as the carrier gas for the vaporization.

5 2. Process according to claim 1, characterized in that the water vapour is employed at a temperature of from 120 to 600°C and preferably from 200 to 550°C.

3. Process according to either of claims 1
10 and 2, characterized in that the aminonitrile is employed at a temperature of from 20 to 300°C and preferably from 100 to 250°C.

4. Process according to one of claims 1 to
15 3, characterized in that the mixture of aminonitrile in the water vapour that is obtained is brought rapidly in a heat exchanger to a temperature at which the vaporization of the mixture is complete.

5. Process according to one of claims 1 to
20 4, characterized in that the mixture of aminonitrile in the water vapour that is obtained is brought to the temperature of reaction between the aminonitrile and water, preferably to a temperature of from 200 to 450°C and, more preferably, from 250 to 400°C.

25 6. Process according to one of claims 1 to 5, characterized in that the aminonitrile is a linear or branched aliphatic aminonitrile having 3 to 12 carbon atoms.

7. Process according to one of claims 1 to 6, characterized in that the aminonitrile originates from the hydrogenation to a primary amine function of one of the two nitrile functions of a dinitrile selected from adiponitrile, methylglutaronitrile, ethylsuccinonitrile, dimethylsuccinonitrile, malononitrile, succinonitrile, glutaronitrile and dodecanedinitrile and is preferably 6-amino-capronitrile.
8. Process according to one of claims 1 to 7, characterized in that the vaporization of the aminonitrile is conducted under an absolute pressure of from 0,1 to 3 bar.
9. Process according to one of claims 1 to 8, characterized in that it is performed with a system without retention of liquid.
10. Process according to claim 9, characterized in that one of the following technologies is employed:
- evaporation of the aminonitrile as a film on a heated surface, in an evaporator of falling-film type;
 - evaporation of an at least partly liquid mist of aminonitrile in the superheated water vapour.
11. Process according to claim 10, characterized in that the distribution of the aminonitrile over the tubes of the falling-film evaporator is carried out by:
- supplying the at least partly liquid

aminonitrile to the tube plate, then distributing this aminonitrile in each tube;

- distributing the at least partly liquid aminonitrile in each tube by atomization to a mist of
- 5 the aminonitrile above the tube plate.

12. Process according to claim 10, characterized in that the evaporation of an at least partly liquid mist of aminonitrile in superheated water vapour is single-stage or multistage.

- 10 13. Process according to one of claims 1 to 12, characterized in that the system for vaporizing the aminonitrile is selected such that the dwell time of liquid aminonitrile in the said system is less than or equal to one minute, preferably less than or equal to 5
- 15 seconds.

PROCESS FOR VAPORIZING AMINONITRILE

The present invention relates to the vaporization of aminonitrile and water under conditions which limit or eliminate the formation of heavy by-products, especially aminocarboxylic acid oligomers.

In order to avoid this drawback, a process for vaporizing aminonitrile and water has now been found which is characterized in that the water, in the vapour state, is used as the carrier gas for the vaporization.

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY
(Includes Reference to Provisional and PCT International Applications)

Attorney's Docket No.

022701-915

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD FOR EVAPORATING AMINONITRILE

the specification of which (check only one item below):

☐ is attached hereto.

☐ was filed as United States application

Number _____

on _____

and was amended

on _____ (if applicable).

☒ was filed as PCT international application

Number PCT/FR99/01524

on 24 June 1999

and was amended

on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 (a)-(e) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §119:

COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §119
FR	98/08258	25 June 1998	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONT'D)
(Includes Reference to Provisional and PCT International Applications)

Attorney's Docket No.

022701-915

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose to the Office all information known to me to be material to the patentability as defined in Title 37, Code of Federal Regulations §1.56, which became available between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. §120:

U.S. APPLICATIONS		STATUS (check one)		
U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED
PCT APPLICATIONS DESIGNATING THE U.S.				
PCT APPLICATION NO.	PCT FILING DATE	U.S. APPLICATION NUMBERS ASSIGNED (if any)		

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

William L. Mathis	17,337	Eric H. Weisblatt	30,505	Bruce T. Wieder	33,815
Robert S. Swecker	19,885	James W. Peterson	26,057	Todd R. Walters	34,040
Platon N. Mandros	22,124	Teresa Stanek Rea	30,427	Ronni S. Jillions	31,979
Benton S. Duffett, Jr.	22,030	Robert E. Krebs	25,885	Harold R. Brown III	36,341
Norman H. Stepno	22,716	William C. Rowland	30,888	Allen R. Batum	36,086
Ronald L. Grudziecki	24,970	T. Gene Dillahunty	25,423	Steven M. duBois	35,023
Frederick G. Michaud, Jr.	26,003	Patrick C. Keane	32,858	Brian P. O'Shaughnessy	32,747
Alan E. Kopecki	25,813	B. Jefferson Boggs, Jr.	32,344	Kenneth B. Leffler	36,075
Regis E. Sluter	26,999	William H. Benz	25,952	Fred W. Hathaway	32,236
Samuel C. Miller, III	27,360	Peter K. Skiff	31,917	Wendi L. Weinstein	34,456
Robert G. Makai	28,531	Richard J. McGrath	29,195	Mary Ann Dillahunty	34,576
George A. Hovancec, Jr.	28,223	Matthew L. Schneider	32,814		
James A. LaBarre	28,632	Michael G. Savage	32,296		
E. Joseph Gess	28,510	Gerald F. Swiss	30,113		
R. Danny Huntington	27,903	Charles F. Wieland III	33,096		



21839

and:

Address all correspondence to:



21839

Norman H. Stepno
BURNS, DOANE, SWECKER & MATHIS, L.L.P.
P.O. Box 1404
Alexandria, Virginia 22313-1404

Address all telephone calls to: Norman H. Stepno at (703) 836-6620.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FULL NAME OF SOLE OR FIRST INVENTOR Gerald BOCQUENET		SIGNATURE <i>G. Bocquet</i>	DATE 03.28.01
RESIDENCE 17, le Verger Sud, F-69360 Communay, FR		CITIZENSHIP FR	
POST OFFICE ADDRESS 17, le Verger Sud, F-69360 Communay, FR			
FULL NAME OF SECOND JOINT INVENTOR, IF ANY Henri CHIARELLI		SIGNATURE <i>Chiarelli</i>	DATE 03.28.01
RESIDENCE Lotissement Vialarzeu, Rue du 30 Mai 1944, F-69360 Communay, FR		CITIZENSHIP FR	
POST OFFICE ADDRESS Lotissement Vialarzeu, Rue du 30 Mai 1944, F-69360 Communay, FR			
FULL NAME OF THIRD JOINT INVENTOR, IF ANY Philippe LECONTE		SIGNATURE <i>Leconte</i>	DATE 03.29.01
RESIDENCE 43, rue Sainte-Beuve, F-69330 Meyzieu, FR		CITIZENSHIP FR	
POST OFFICE ADDRESS 43, rue Sainte-Beuve, F-69330 Meyzieu, FR			
FULL NAME OF FOURTH JOINT INVENTOR, IF ANY		SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			
FULL NAME OF FIFTH JOINT INVENTOR, IF ANY		SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			
FULL NAME OF SIXTH JOINT INVENTOR, IF ANY		SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			
FULL NAME OF SEVENTH JOINT INVENTOR, IF ANY		SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			
FULL NAME OF EIGHTH JOINT INVENTOR, IF ANY		SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			